

	<b>International Wool Textile Organisation I.W.T.O.</b>
	<b>Florence Meeting June 1999</b>

# **Technology and Standards Committee**

## **Commercial Technology Forum**

**Chairman : D.J. Ward (Australia)**

**Techn. Coordinator: G. Mercier (France)**

**Report Nrº CTF 04**

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# INTERNATIONAL WOOL TEXTILE ORGANISATION

## TECHNOLOGY & STANDARDS COMMITTEE

## FLORENCE MEETING

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Chairman: D.J. WARD (Australia)

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### Industrial Use of Liposomes in Wool Dyeing

By

L. Coderich, M. Marti, A. De La Maza, A. M. Manich, S. Serra\*, J.M. Fiadeiro\*\* and J.L. Parra

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## BRIEFING PAPER

### SUMMARY OF FINDINGS

Liposome is considered as a new biocompatible product more suitable and promising than conventional synthetic auxiliaries to reach a new clean dyeing technology. A comparative methodology has been used dyeing wool and wool blends with conventional auxiliaries or with a commercial Liposome (Ecotrans W-8814) to check three main parameters: the quality of the dyed textile, the energy saving and ecological impact of the new process.

The usefulness of Liposomes to dye wool and wool blends presented in raw, top material and spun yarn is indicated. The dyeing temperature could be 10°C lower than conventional dyeing, with a dye exhaustion of the same level and a lower environmental impact. However, this Liposome does not favour dye migration to achieve good leveling for woven fabric without presence of synthetic auxiliaries. Design of new Liposome formulations is envisaged.

### COMMERCIAL IMPLICATIONS – CURRENT AND FUTURE

The design of industrial process using milder experimental conditions (e.g. lower temperatures, lower contamination, etc.) still remains one of the most important needs for the textile field. At present, this new process for wool dyeing is being investigated by 13 partners from Portugal, Italy and Spain in an EC Project. Several wool presentations (raw material, top, spun yarn, woven and knitted fabric) are considered and some other textile materials (polyester, viscose, polyamides, and acrylics) are also envisaged in blends with wool.

This process may be considered a new clean technology approach already used by some Textile Industries. The final economical optimisation of the dyeing process using Liposomes imply:

- an improvement of quality of the wool dyed in terms of leveling, dye fixation, wool smoothness, tensile strength and better weight yield of the wool processed.
- economical advantages in terms of Liposome cost, dyestuff cost (higher exhaustion in the bath) and energy saving (the dyeing process may be performed at 85-90°C instead of the conventional temperature 98°C).
- a lower ecological impact of the dyeing process, since the use of Liposomes, from biological origin, instead of synthetic dyeing auxiliaries may permit a clear reduction of the wastewater negative charge.

### NOTE

This paper will be discussed at the Commercial Technology Forum, at 0900 on Sunday 30<sup>th</sup> May, 1999.

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## SUMMARY

Liposome is considered as a new biocompatible product more suitable and promising than conventional synthetic auxiliaries to reach a new clean dyeing technology. A comparative methodology has been used dyeing wool and wool blends with conventional auxiliaries or with a commercial Liposome (Ecotrans W-8814) to check three main parameters: the quality of the dyed textile, the energy saving and ecological impact of the new process.

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## INTRODUCTION

Over the last decade, a number of investigations have been carried out using different carriers capable of reducing the degradation brought about in conventional wool dyeing. At present, there is a commercial Liposome in the textile market, Ecotrans W-8814, with competitive cost to conventional auxiliaries. This new dyeing auxiliary has been demonstrated in the laboratory to control the rise of the colour giving up to good levelness with the advantage to promote higher dye exhaustion than other synthetic auxiliaries. Furthermore, the final temperature of the dye process can be reduced on 10°C, favouring the handle characteristics of the fibre with energy saving. The biological nature of the Liposome is an important ecological benefit of this new dyeing process.

To demonstrate the industrial feasibility of this dyeing process, 13 Industries participate in an European project comparing their particular dyeing process with the one in presence of Liposome. In the present work some of these experiences are presented and the processes are evaluated in terms of quality of the textile dyed, energy saving and ecological impact of the new process.

As regards to Quality, an improvement of the dye exhaustion and dye migration onto the fibre is the main goal. It has been demonstrated this parameter mainly in the dyeing of wool and other natural fibres (angora, mohair, silk, etc) at a raw material and spun yarn presentations. The main reasons for this better performance are: The use of liposomes as an appropriate vehicle, their biological characteristics, the possibility to reduce the final temperature of the dyeing bath avoiding a deleterious effect on the fibre, etc. Moreover, the use of liposomes during the dyeing of wool (raw material or top) increases the industrial yield of the subsequent spinning, weaving and knitting processes (3-5%).

As regards to energy saving, this innovative process permits a reduction of about 10°C in the final dyeing temperature resulting in an 8-10% energy saving. Reduction of the Chemical Oxygen Demand (COD) in the wastewater (about 1000 units) of the dyeing bath is another important benefit. About 2 Euro reduction of the cost of wastewater treatment per 1 TM of wool dyed may be expected. Furthermore, the cost of liposomes is similar or cheaper (about 3.5 - 4 Euro/l) than the cost of conventional auxiliaries (about 4.5 Euro/l).

However, the wool industry demands a new generation of liposomes able to be used in the dyeing of wool fabrics submitted previously to different chemical treatments during their processing before the final dyeing. In particular, it could be very convenient to improve the dye migration phenomena. This is an important industrial objective that we will try to manage in the future designing new strategic Liposome formulations.

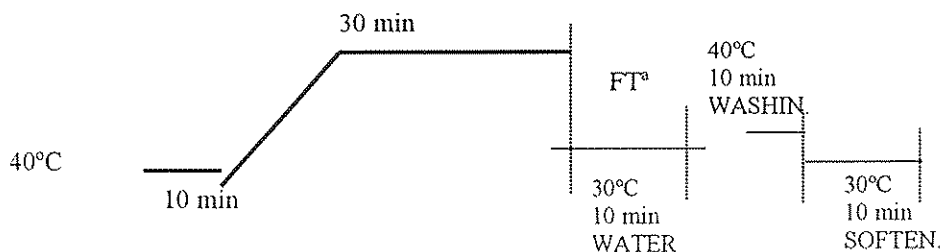
## **DYEING OF WOOL AT DIFFERENT PRESENTATION FORMS**

### **RAW AND TOP WOOL**

Raw material dyeing has been performed using *pre-metallised 2:1 dyes (Lanaset dyes)* with 0.7% owf of Ecotrans W-8814 Liposome, at a final temperature of 90°C while in the case of conventional dyeing 0.3% owf of Tanapal and 1% owf of Tanaterge NWA, at a final temperature of 98°C was applied (Table 1).

Table 1. Dyeing recipes and ecological impact analysis of raw wool

	<b>LIPOSOME DYEING</b>	<b>CONVENTIONAL DYEING</b>
Liquor ratio	1: 9.5	
Dyes	Yellow Lanaset 2R: 0.075 % owf Brown Lanaset B: 0.071 % owf Brown Lanaset G: 0.048 % owf	
Textile auxiliary	Ecotrans W-8814: 0.7 % owf Acetic Acid 80%: 1 % owf Formic Acid 85%: 0.5 % owf	Tanapal LM: 0.5 % owf Tanaterge NWA: 1% owf Acetic Acid 80%: 1 % owf Formic Acid 85%: 0.5 % owf
<b>Ecological impact analysis</b>		
Dye Exhaustion (%)	95	88
COD (mgO <sub>2</sub> /l)	2205	3850
BOD (mgO <sub>2</sub> /l)	671	996
COD/BOD	3.28	3.8
Conductivity (mS/cm) and SOL (µS/cm)	2.72 3489	4.53 5945
PH	No influence	No influence
Phosphor (mg phosphor total/l)	3	8
Nitrogen ammoniac (mg NH <sub>4</sub> <sup>+</sup> /l)	12	160

**Dyeing Cycle**

FT=90°C for Liposome dyeing, FT=98°C for Tanapal dyeing

In Table I is possible to appreciate a better behaviour of Liposome dyeing compared with Tanapal dyeing. When Liposome is present in the dye-bath the dye exhaustion increases, and environmental parameters decrease.

The material weight balance of the wool samples dyed in the presence or absence of liposomes during the subsequent textile processing steps were evaluated. Part of the raw material dyed using liposomes (1700 Kg) was spun in an industrial process and it was compared with a raw material dyed with conventional auxiliaries.

The results attained were satisfactory. A very high yield was obtained, 97.46% and the physical properties evaluated gave the following results, tenacity: 4.49 CN/TEX, elongation: 17.83% and maximum friction force: 347.8 CN (6% higher than the ones obtained in conventional dyeing).

It is important to point out that till now 70 tons of raw material and top wool have been industrially satisfactorily dyed with 32 different dyes.

**SPUN YARN**

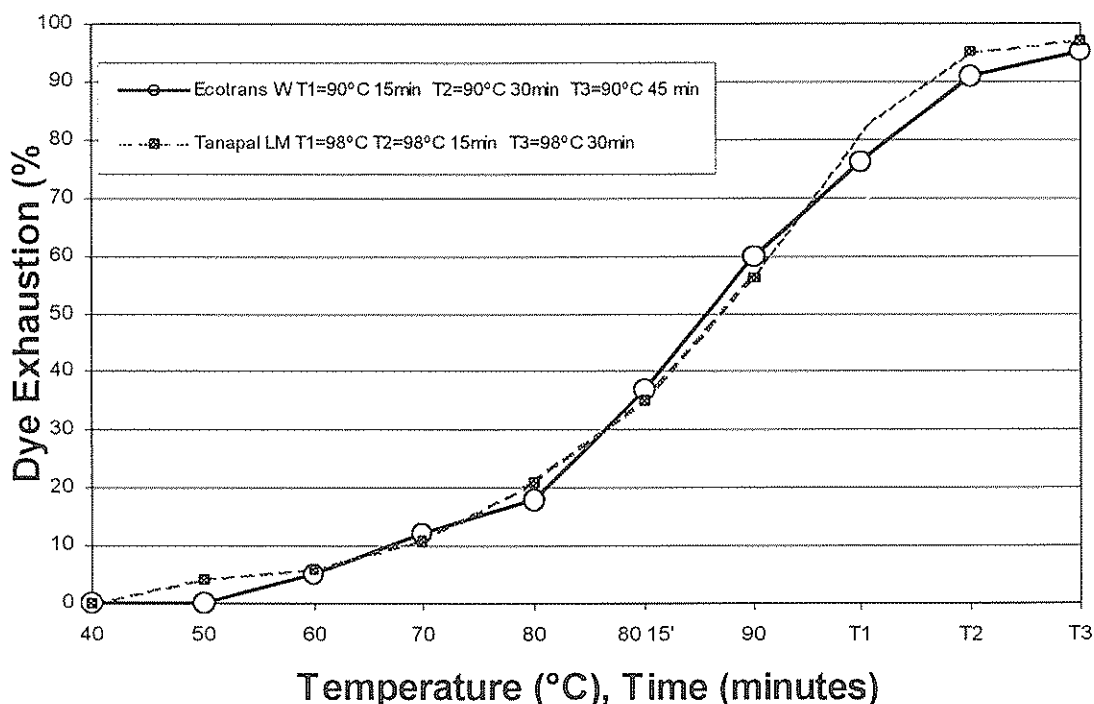
Worsted Yarn material (one single strand) was dyed using pre-metallised 2:1 dyes (Lanaset dyes) with 1% owf of Ecotrans W-8814 Liposome, at a final temperature of 90°C and with the industrial process, in presence of a 1% owf of Tanapal LM at a final temperature of 98°C. The similar kinetic profile obtained bearing in mind the lower temperature in the Liposome process also supports the use of this Liposome in preference to Tanapal LM.

**Dyeing Recipe**

	LIPOSOME DYEING	CONVENTIONAL DYEING
Liquor ratio	1: 10	
Dyes	Grey Lanaset G: 1.235 % owf Orange Lanaset RN: 0.795% owf Navy Blue Lanaset R: 0.730% owf Navy Blue Lanaset SDNLS: 0.72% owf Blue Polyamid CBL 150: 0.345% owf Red Lanaset G: 0.055% owf	
Textile auxiliary	Emectol SMA:0.2 g/l	Emectol SMA:0.2 g/l
	Ecotrans W-8814: 1% owf	Tanapal LM:1 % owf
	Acetic Acid 80%: pH=4.5-5	Acetic Acid 80%: pH=4.5-5

Posterior treatment

Posterior Washing	Hostapon T dust a. c.: 0.2% owf
Softening Treatment	Steramine PNA 160: 1% owf Formic Acid 85%:0.3% owf

Dye exhaustion of Pre-metallised 2:1 Dye

A similar kinetic profile was obtained bearing in mind the lower temperature in the case of Liposome process compared to the Tanapal LM process. Also, spun yarn of Shetland wool (two plied yarn) was dyed using either Ecotrans W-8814 or Tanapal LM to measuring mechanical properties.

Mechanical properties of wool processed were evaluated to evidence the quality improvement imparted to the wool samples dyed by liposomes in comparison with the wool dyed by a conventional procedure.

Mechanical properties and characteristics have been measured on raw yarns and dyed yarns using conventional auxiliaries, and liposomes. Table II shows the results obtained when linear density and twist have been measured on yarns. As it can be expected there were not significant differences between these parameters measured on yarns dyed with auxiliaries and yarns dyed with liposomes. Yarn linear density was measured on a sample of 100 m of yarn and twist was assessed as a mean value of 30 samples of 50 cm of length using the conventional twist-untwist method.

Table II. Results on linear density and twist.

	No. (Tex)	Twist (v/m)
Shetland Wool (two plied yarn)		
Untreated wool	231.41	65.26 S
Tanapal LM	211.13	59.63 S
Ecotrans W-8814	214.24	59.20 S
Wool Yarn 2/43 (one single strand yarn)		
Untreated wool	30.80	466.53 S
Tanapal LM	30.34	497.20 S
Ecotrans W-8814	31.10	494.96 S

Load and Elongation to break were determined on 30 samples of yarn 250 mm of length, submitted to a traction test at a 60%/min gradient of stretching (150 mm/min), according to the conditions shown in the ASTM Standard D 885 with some modifications. Breaking load [daN], Tenacity [cN/tex] and Elongation at Break were given as a mean value. Also the coefficient of variation were calculated. Results are shown in Table III. No significant differences between results were observed. However, higher values were always obtained in the case of Liposome related to the Tanapal LM dyed sample.

Table III. Results on mechanical properties.

Yarn Breaking Tests			
	Break. Load daN / C.V.%	Tenacity cN/tex / CV%	Elongation % / CV%
Shetland Wool (two plied yarn)			
Untreated wool	0.76 / 4.4	3.29 / 4.4	15.00 / 9.1
Tanapal LM	0.61 / 8.4	2.87 / 8.4	10.15 / 12.9
Ecotrans W-8814	0.64 / 7.5	2.99 / 7.5	10.23 / 26.5
Wool Yarn 2/43 (one single strand yarn)			
Untreated wool	0.23 / 10.6	7.57 / 10.6	17.73 / 19.6
Tanapal LM	0.21 / 10.6	6.91 / 10.6	15.47 / 23.8
Ecotrans W-8814	0.21 / 10.4	6.92 / 10.4	18.57 / 25.5

Handle assessment were done through the determination of yarn to yarn friction coefficient and yarn to metal friction coefficient according to the ASTM Standards D 3108 and D 3412 respectively with some modifications Results are shown in Table IV.

Yarn to yarn friction shows that yarn dyed with Ecotrans has a handle more similar to the raw wool yarn than yarn dyed with Tanapal. Considering yarn to metal friction, when Ecotrans were used as auxiliary in the case of two plied yarn, the handle were more similar to the raw wool yarn than when Tanapal was used. In the case of one single strand yarns, there were no differences between Ecotrans and Auxiliary dyeing.

Table IV. Results on friction.

	Yarn to Yarn	Yarn to Metal
Shetland Wool (two plied yarn)		
Untreated wool	75.20	65.68
Tanapal LM	66.60	50.08
Ecotrans W-8814	77.60	61.52
Wool Yarn 2/43 (one single strand yarn)		
Untreated wool	27.44	35.30
Tanapal LM	22.00	34.56
Ecotrans W-8814	23.68	34.24

All parameters considered leads to a conclusion that Liposome dyeing gives no significant differences in relation to the conventional auxiliary dyeing. Other measurements like yield during weaving and mechanical properties and handle of woven fabrics are planned to be performed in the future.

### WOOL YARN WITH SUPERWASH TREATMENT

Wound packages cones of worsted yarn, Nm 29, 100%wool with Superwash treatment was submitted to dyeing process with metal complex dyes 1:2 by the conventional and Liposome methods.

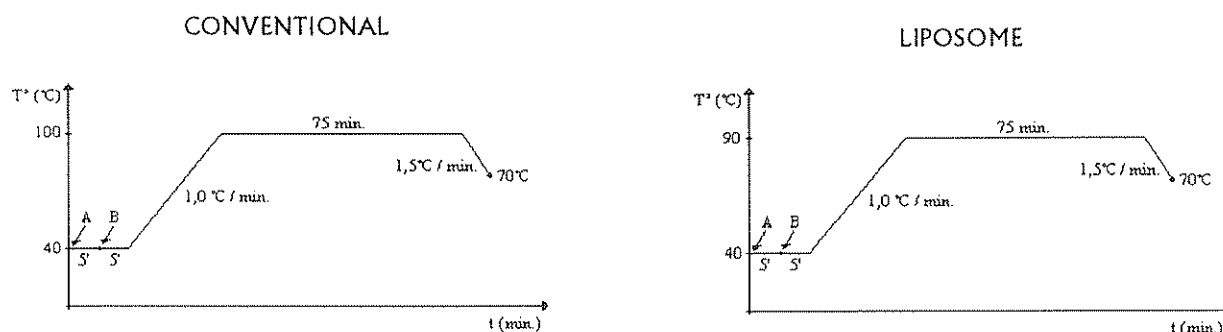
#### DYEING CONDITIONS:

	Recipes		
	CONVENTIONAL	LIPOSOME 1	LIPOSOME 2
<b><u>A – Auxiliaries:</u></b>			
Scourol S-520	0,5 g/l	----	-----
ECO TRANS W - 8814	----	0,8 % o.w.f.	0,8 % o.w.f.
Protintex 1779/P Conc.	1,0 % o.w.f.	----	-----
Protisol STA	1,5 % o.w.f.	----	-----
Protintex HR	1,0 g/l	----	-----
Avolan UL 75	----	----	1,0 % o.w.f.
Ammonium Sulphate	3,0 % o.w.f.		3,0 % o.w.f.
Sodium Sulphate	----		2,0 g/l
Eulisin S	0,8 % o.w.f. (pH: 5,0)	0,8 % o.w.f. (pH: 5,0)	0,8 % o.w.f. (pH: 5,0)
<b><u>B – ACIDOL dyestuffs:</u></b>			
MBL Brown	0,9 % o.w.f.	0,9 % o.w.f.	0,9 % o.w.f.
GNW Yellow	0,1 % o.w.f.	0,1 % o.w.f.	0,1 % o.w.f.
MTR dark blue	0,7 % o.w.f.	0,7 % o.w.f.	0,7 % o.w.f.

R.B.: 15 l/Kg



## DYEING DIAGRAM:



## RESULTS:

		CONVENTIONAL	LIPOSOME 1	LIPOSOME 2
COD initial bath (mg/l O <sub>2</sub> )		2895	2395	2327
BOD <sub>5</sub> initial bath (mg/l O <sub>2</sub> )		777	460	495
COD final bath (mg/l O <sub>2</sub> )		2542	1420	1625
BOD <sub>5</sub> final bath (mg/l O <sub>2</sub> )		545	400	420
Tensile strength of undyed yarn (cN)		186±6		
Coefficient of variation (%)		12,12		
Tensile strength after dyeing (cN)		201±6	186±7	210±6
Coefficient of variation (%)		10,24	14,45	11,1
Colour fastness to washing (ISO 105 C06 A2S)	Colour change	4-5	4	4
	Staining (WO/PA/CO)	4-5 / 4 / 4-5	4-5 / 4 / 4-5	4-5 / 4 / 4-5
Colour fastness to water (ISO 105 E01)	Colour change	4-5	4-5	4-5
	Staining (WO/PA/CO)	4-5 / 4-5 / 4-5	4-5 / 3-4 / 4-5	4-5 / 4-5 / 4-5
Dyebath Exhaustion (%)		89,10	99,01	88,20
Optical density of final bath (%)		1,15633	1,15077	-----
Colour levelness		GOOD	POOR	GOOD
Costs (PTE/Kg): and auxiliaries	- Dyes	96,03	67,55	79,93
	- Electrical energy	17,62	15,91	15,91
	- Thermal energy	21,06	14,45	14,45
	Total	126,39	97,91	110,29

Dyeing with liposomes under acid conditions similar to those used in conventional process (Recipe 1) was not satisfactory, about 80% of the dyestuff being exhausted onto the fibres after 5 min. at 40°C and consequently a pronounced dyeing unevenness between the inside and the outside of the cones was produced.

In an attempt to reduce the dyeing rate and to improve evenness some further experiments employing a neutral bath (pH≈ 6,8) and a low temperature at the beginning of the dyeing process (30°C) were then carried out but these conditions had a very little effect on the results, even when the temperature was maintained at 30°C during 30 min. It therefore was decided to employ a retarding agent (AVOLAN UL 75). It was found that an add-on level of 1% of this agent and 2g/l of sodium sulphate (Recipe 2) reduced the dyeing to an acceptable level, similar to that of the conventional dyeing.

The colour evenness was increased significantly, the dyeing costs being still lower than those of conventional dyeing.

From the tabled data the following can be observed for Liposome – leveling agent dyeing:

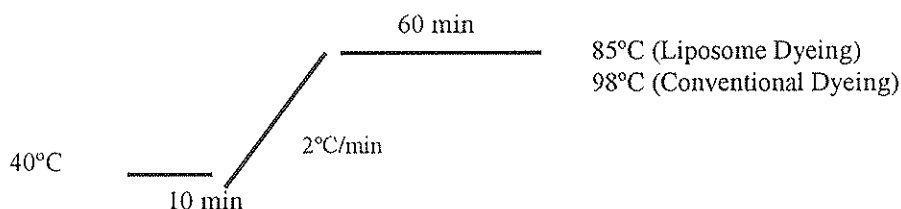
- A slightly higher tensile strength of the dyed yarn;
- A slightly higher change of colour concerning fastness to washing (difference of a half-step);
- A polluting charge reduction of about 40% for COD and 20% for BOD<sub>5</sub>;
- A reduction of about 12% for dyeing costs.

## WOVEN WOOL FABRIC

Woven fabric dyeing has been made using *pre-metallised 1:1 dyes (Neolan dyes)*. In this case two different kinetics were compared, first with conventional auxiliaries using a final temperature of 98°C and second with Liposome Ecotrans W-8814 instead of sodium sulphate at 85°C of final temperature. In this case, the presence of conventional auxiliaries such as Matexil and Avolan are necessary to achieve even dyeing.

Liposomes mainly restrain the rise of colour at low temperatures without a pronounced action as migrator. Woven fabrics have suffered previous chemical treatments that need a strong migrator to achieve even dyeing. Therefore, in this case the salt that mainly retains the raise of colour has been substituted by Liposome, being necessary the presence of conventional migrators to achieve a high quality of the textile dyed.

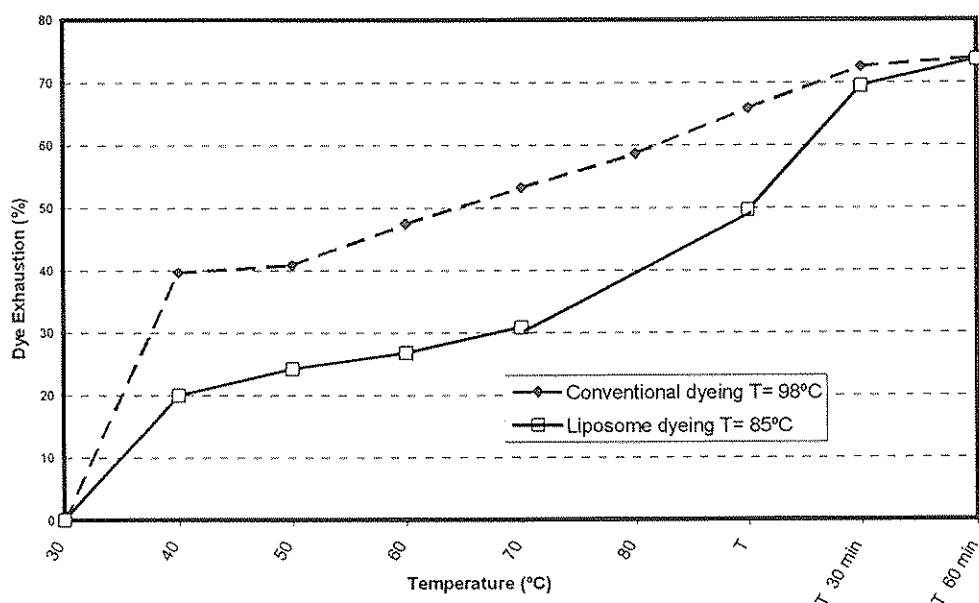
## Dyeing Cycle



## Dyeing Recipe

	LIPOSOME DYEING	CONVENTIONAL DYEING
Liquor ratio	1: 12	
Dyes	Yellow Stenamina GR 175: 0.174 % owf Burdeos Premelan RM: 0.242 % owf Blue Cocelan 2G 250: 0.50 % owf Yellow Neolan RE 250: 0.176 % owf	
Textile auxiliary	Liposome: 0.2 % owf Matexil Wash: 0.5 % owf Avolan SCN: 0.3 % owf Nofelt WA: 1.5 % owf Sulfuric Acid: 3 % owf	Sodium Sulphate: 7.5 owf Matexil Wash: 0.5 % owf Avolan SCN: 0.3 % owf Nofelt WA: 1.5 % owf Sulfuric Acid: 3 % owf

## Dyeing kinetics of woven fabric



It can be observed the higher controlled dye absorption during the dyeing in presence of liposomes, being the final exhaustion similar for both dyeing. Furthermore, the final temperature of the Liposome dyeing process can be reduced on 13°C that will greatly favours the handle characteristics of the woven fabric with an additional energy saving.

## DYEING OF WOOL BLENDS

Wool/Polyamide yarns were dyed with metal complex dyes 1:2, metal complex dyes 1:1 and milling acids dyes all by the conventional and Liposome methods (cone dyeing).

Acrylic/wool yarns were dyed with cationic and metal complex dyes 1:2 (one-bath; two-step process) by the conventional and Liposome methods (cone dyeing). Wool/Acrylic/Alpaca yarns were dyed with metal complex dyes 1:2 for wool (1<sup>st</sup> bath) and cationic dyes for acrylic (2<sup>nd</sup> bath) by the conventional and the Liposome methods (hank dyeing).

For each test the dye-bath exhaustion, the polluting load, the mechanical strength of the yarn before and after dyeing, the colour levelness, the fastness properties of dyeings and specific costs relating to dyestuffs and auxiliaries, electric power and thermic energy (vapour) were evaluated.

### 1. WOOL/POLYAMIDE DYEINGS

#### 1.1 DYEING OF WOOL/POLYAMIDE BLENDS USING METAL COMPLEX DYES 1:2

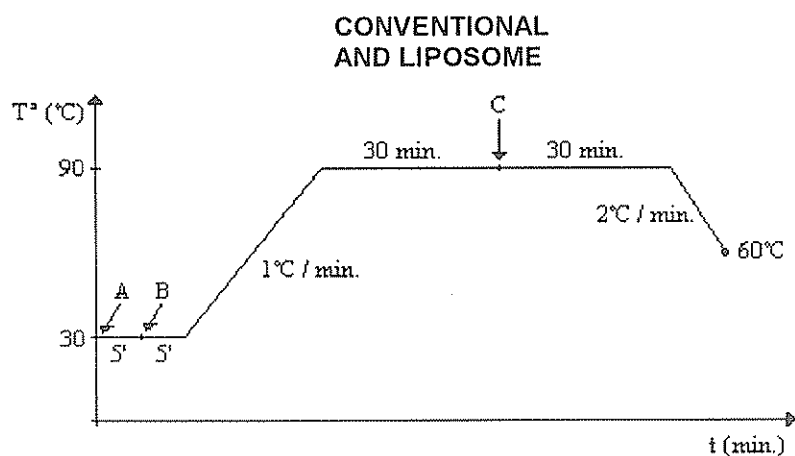
Wound packages (cones) of woollen yarn, Nm 12, 70% wool 30% Polyamide (intimate blend)

## DYEING CONDITIONS:

	Recipes	
	CONVENTIONAL	LIPOSOME
<b>A – Auxiliaries :</b>		
Edolan PAW Líq.	1,0 % o.w.f.	1,0 % o.w.f.
Baylan CNT	2,0 % o.w.f.	-----
Avolan UL 75	0,5 % o.w.f.	-----
ECO TRANS W - 8814	-----	0,8 % o.w.f.
Sodium Sulphate	1,0 g/l	-----
Tri-Sodium Phosphate	0,5 g/l	-----
Acetic Acid	1,0 g/l (pH: 4,5)	1,0 g/l (pH: 4,5)
<b>B – Dyestuffs:</b>		
ISOLAN Yellow SGL	0,30 % o.w.f.	0,30 % o.w.f.
ISOLAN Bordeaux SBL	2,40 % o.w.f.	2,40 % o.w.f.
ISOLAN Dark blue 2SGL	0,22 % o.w.f.	0,22 % o.w.f.
<b>C :</b>		
Formic Acid	(Ph: 3,8 - 4,0)	(pH: 3,8 - 4,0)

R.B.: 10 l/Kg

## DYEING DIAGRAM:



**RESULTS:**

		CONVENTIONAL	LIPOSOME
COD initial bath (mg/l O <sub>2</sub> )		14172	12068
BOD <sub>5</sub> initial bath (mg/l O <sub>2</sub> )		3315	2665
COD final bath (mg/l O <sub>2</sub> )		7469	7278
CBO <sub>5</sub> final bath (mg/l O <sub>2</sub> )		2145	2600
Tensile strength of undyed yarn (cN)		399±8	
Coefficient of variation (%)		12,3	
Tensile strength after dyeing (cN)		291±8	290±4
Coefficient of variation (%)		17,3	15,2
Colour fastness to washing (ISO 105 C06 A2S)	Colour change	4-5	4-5
	Staining (WO/PA/CO)	4-5 / 4-5 / 4-5	4-5 / 4-5 / 4-5
Colour fastness to water (ISO 105 E01)	Colour change	4-5	4-5
	Staining (WO/PA/CO)	4-5 / 4-5 / 4-5	4-5 / 4-5 / 4-5
Dyebath Exhaustion (%)		97,7	97,8
Optical density of final bath (%)		1,1051	1,00
Colour levelness		GOOD	GOOD
Costs (PTE/Kg): - Dyes and auxiliaries		192,80	170,71
- Electrical energy		5,50	5,50
- Thermal energy		8,60	8,60
Total		206,90	184,81

**1.2 WOOL/POLYAMIDE YARN, DYED WITH METAL COMPLEX DYES 1:1**

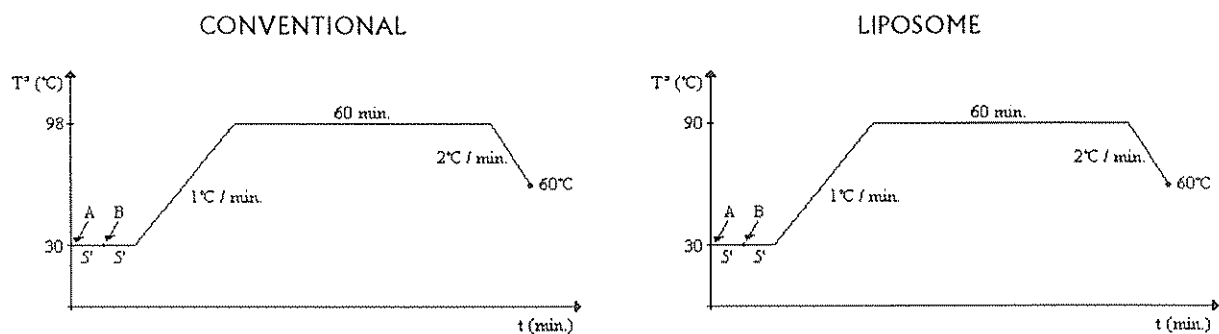
Wound packages of woollen yarn, Nm 12, 70%Wool/30%Polyamide (intimate blend)

**DYEING CONDITIONS:**

	Recipes	
	CONVENTIONAL	LIPOSOME
<b>A – Auxiliaries:</b>		
Keriolan A2N	1,5 % o.w.f.	-----
ECO TRANS W - 8814	-----	0,8 % o.w.f.
Sodium Sulphate	10,0 % o.w.f.	-----
Sulphuric Acid	5,0 % o.w.f. (pH: 2)	5,0 % o.w.f. (pH: 2)
<b>B – NEOLAN dyestuffs:</b>		
RE Yellow 250%	0,30 % o.w.f.	0,30 % o.w.f.
RM Bordeaux 200%	2,40 % o.w.f.	2,40 % o.w.f.
3R Blue 200%	0,22 % o.w.f.	0,22 % o.w.f.

R.B.: 20 l/Kg

## DYEING DIAGRAMS:



## RESULTS:

		CONVENTIONAL	LIPOSOME
COD initial bath (mg/l O <sub>2</sub> )		2091	1601
BOD <sub>5</sub> initial bath (mg/l O <sub>2</sub> )		n. d.	n. d.
COD final bath (mg/l O <sub>2</sub> )		1585	830
BOD <sub>5</sub> final bath (mg/l O <sub>2</sub> )		163	100
Tensile strength of undyed yarn (cN)		399±8	
Coefficient of variation (%)		12,3	
Tensile strength after dyeing (cN)		415±8	444±9
Coefficient of variation (%)		11,8	12,0
Colour fastness to washing (ISO 105 C06 A2S)	Colour change	3-4	3-4
	Staining (WO/PA/CO)	4-5 / 4-5 / 4	4-5 / 4-5 / 4
Colour fastness to water (ISO 105 E01)	Colour change	4-5	4-5
	Staining (WO/PA/CO)	3-4 / 2-3 / 3-4	3 / 2 / 3
Dyebath Exhaustion (%)		91,8	94,0
Optical density of final bath (%)		1,1879	0,7496
Colour levelness		GOOD	GOOD
Costs (PTE/Kg):	- Dyes and auxiliaries	168,46	156,91
	- Electrical energy	6,00	5,50
	- Thermal energy	22,86	17,20
Total		197,32	179,61

### 1.3 WOOL/POLYAMIDE YARN, DYED WITH MILLING ACID DYES

Wound packages (cones) of woollen yarn, Nm 12, 70%Wool/30%Polyamide (intimate blend)

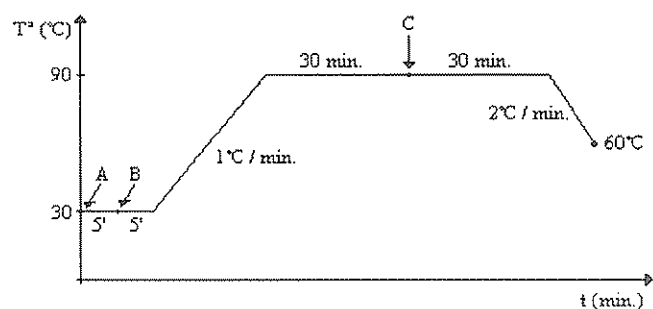
#### DYEING CONDITIONS:

	Recipes	
	CONVENTIONAL	LIPOSOME
<b>A – Auxiliaries :</b>		
Edolan PAW Líq.	0,8 % o.w.f.	0,8 % o.w.f.
Baylan CNT	2,0 % o.w.f.	-----
Avolan UL 75	0,5 % o.w.f.	-----
ECO TRANS W - 8814	-----	0,8 % o.w.f.
Sodium Sulphate	1,0 g/l	-----
Tri-Sodium Phosphate	0,5 g/l	-----
Acetic Acid	1,0 g/l (pH: 4,5)	1,0 g/l (pH: 4,5)
<b>B – Dyestuffs:</b>		
SUPRANOL Yellow SWP	0,50 % o.w.f.	0,50 % o.w.f.
SUPRANOL Red SWP	0,50 % o.w.f.	0,50 % o.w.f.
SUPRANOL Blue SWP	2,50 % o.w.f.	2,50 % o.w.f.
<b>C :</b>		
Formic Acid	(pH: 3,8 - 4,0)	(pH: 3,8 - 4,0)

R.B.: 10 l/Kg

#### DYEING DIAGRAM:

##### CONVENTIONAL AND LIPOSOME



**RESULTS:**

		CONVENTIONAL	LIPOSOME
COD initial bath (mg/l O <sub>2</sub> )		14085	9245
BOD <sub>5</sub> initial bath (mg/l O <sub>2</sub> )		2254	2120
COD final bath (mg/l O <sub>2</sub> )		6538	5463
BOD <sub>5</sub> final bath (mg/l O <sub>2</sub> )		2251	1401
Tensile strength of undyed yarn (cN)		399±8	
Coefficient of variation (%)		12,3	
Tensile strength after dyeing (cN)		228±8	279±8
Coefficient of variation (%)		17,8	14,5
Colour fastness to washing (ISO 105 C06 A2S)	Colour change	4-5	4-5
	Staining (WO/PA/CO)	4-5 / 3-4 / 4-5	4-5 / 3-4 / 4-5
Colour fastness to water (ISO 105 E01)	Colour change	4-5	4-5
	Staining (WO/PA/CO)	4-5 / 4 / 4-5	4-5 / 4 / 4-5
Dyebath Exhaustion (%)		99,03	99,12
Optical density of final bath (%)		0,46675	0,40196
Colour levelness		EXCELLENT	EXCELLENT
Costs (PTE/Kg): auxiliaries		276,27	254,18
- Dyes and			
- Electrical energy		5,50	5,50
- Thermal energy		8,60	8,60
Total		290,37	268,28

The surface uniformity of the colour, evaluated by visual examination of knit samples made with dyed yarns is identical in conventional and Liposome dyeing. The bath exhaustion is quite high in both methods, no significant differences were found in the values obtained for each one when 1:2 metal complex dyes and milling acid dyes are used. In the case of metal complex dyes 1:1 the % exhaustion achieved by Liposome method is slightly higher.

The polluting load of Liposome exhaustion baths is quite lower than in conventional dyeing process, the differences ranging from 3% to 8% for COD and from 10% to 38% for BOD<sub>5</sub>. The high values of COD and BOD<sub>5</sub> for dyeings with 1:2 metal complex dyes in both processes may result from the use of a retarding agent for polyamide.

Good washing and water fastness levels were achieved, the colour change and staining values being identical for conventional and Liposome dyeings when using 1:2 metal complex dyes or acid milling dyes. From Liposome dyeings with 1:1 metal complex dyes a slightly higher staining equivalent to a half-step was found concerning the fastness to water, the results being identical to those of conventional dyeings concerning the fastness to washing.

No significant differences are found concerning the strength of dyed yarn by both processes, using 2:1 metal complex dyes at the temperature of 90°C. However, when dyeing with acid milling dyes at the same temperature a slightly higher strength was achieved for the Liposome dyed yarn. Dyeing with 1:1 metal complex dyes, at 98°C in the conventional process and at 90°C with Liposome gives up to a higher tensile strength for the yarns submitted to the Liposome process.

The total dyeing costs are about 7% to 11% lower in the Liposome process.



## 2. WOOL/ACRYLIC, WOOL/ACRYLIC/ALPACA DYEINGS

## 2.1 Acrylic/wool yarn, dyed with cationic and metal complex dyes 1:2 (one-bath; two-step process)

Wound packages (cones) of worsted yarn, Nm 2/35, 75%Acrylic/25%Wool (intimate blend)

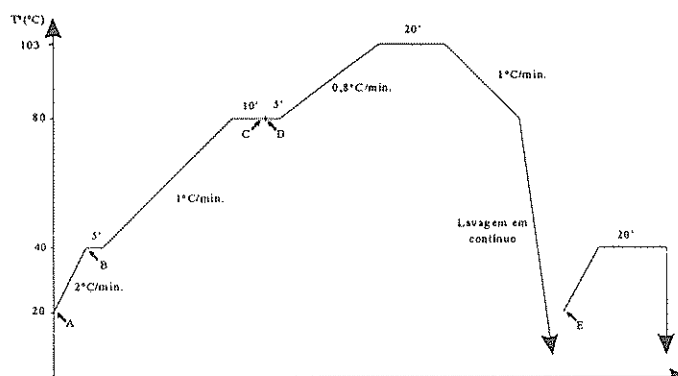
## DYEING CONDITIONS:

	Recipes	
	CONVENTIONAL	LIPOSOME
<b><u>A – Wool Auxiliaries:</u></b>		
Avolan UL 75	1,0 % o.w.f.	-----
ECO TRANS W - 8814	-----	0,8 % o.w.f.
Sodium Acetate	2,0 % o.w.f.	-----
Acetic Acid	2,0 % o.w.f. (pH: 4,5)	2,0 % o.w.f. (pH: 4,5)
<b><u>B – Wool dyestuffs:</u></b>		
ISOLAN Orange SRL	0,0844 % o.w.f.	0,0844 % o.w.f.
SUPRANOL Green BW	0,0364 % o.w.f.	0,0364 % o.w.f.
SUPRANOL Blue RLW	0,1600 % o.w.f.	0,1600 % o.w.f.
SUP. BL Rouge 200%	0,0137 % o.w.f.	0,0137 % o.w.f.
<b><u>C – Acrylic Auxiliaries:</u></b>		
Tubacryl RI	0,80 % o.w.f.	0,80 % o.w.f.
Astragal ME	1,75 % o.w.f.	1,75 % o.w.f.
<b><u>D – Acrylic dyestuffs:</u></b>		
VIOCRYL GRSL Yellow 200%	0,0635 % o.w.f.	0,0635 % o.w.f.
VIOCRYL GGS Red 200%	0,0020 % o.w.f.	0,0020 % o.w.f.
ASTRAZON 2RN Dark Blue	0,1209 % o.w.f.	0,1209 % o.w.f.
<b><u>E – Softening:</u></b>		
Belfasin OET	6,5 % o.w.f.	6,5 % o.w.f.
Acetic Acid	0,87 % o.w.f.	0,87 % o.w.f.

R.B.: 10 l/Kg

## DYEING DIAGRAM:

## CONVENTIONAL AND LIPOSOME



## RESULTS:

		CONVENTIONAL	LIPOSOME
COD initial wool bath (mg/l O <sub>2</sub> )		4016	1680
BOD <sub>5</sub> initial wool bath (mg/l O <sub>2</sub> )		1700	940
COD final wool bath (mg/l O <sub>2</sub> )		3937	1856
BOD <sub>5</sub> final wool bath (mg/l O <sub>2</sub> )		1625	900
COD initial acrylic bath (mg/l O <sub>2</sub> )		4277	3052
BOD <sub>5</sub> initial acrylic bath (mg/l O <sub>2</sub> )		1175	730
COD final acrylic bath (mg/l O <sub>2</sub> )		3694	1758
BOD <sub>5</sub> final acrylic bath (mg/l O <sub>2</sub> )		1300	880
Tensile strength of undyed yarn (cN)		590,72	
Coefficient of variation (%)		6,65	
Tensile strength after dyeing (cN)		558,05	569,41
Coefficient of variation (%)		7,95	7,14
Colour fastness to washing (ISO 105 C06 A2S)	Colour change	5	5
	Staining (WO/PA/CO)	5 / 5 / 5	5 / 5 / 5
Colour fastness to water (ISO 105 E01)	Colour change	5	5
	Staining (WO/PA/CO)	5 / 5 / 5	5 / 5 / 5
Colour levelness		GOOD	EXCELLENT
Costs (PTE/Kg): auxiliaries  energy	- Dyes and	78,92	58,06
	- Electrical	18,32	18,32
	- Thermal energy	13,38	13,38
Total		110,62	89,76

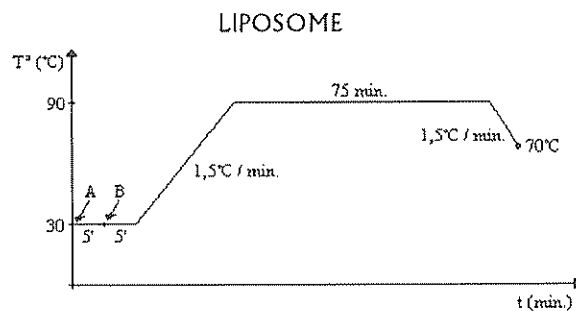
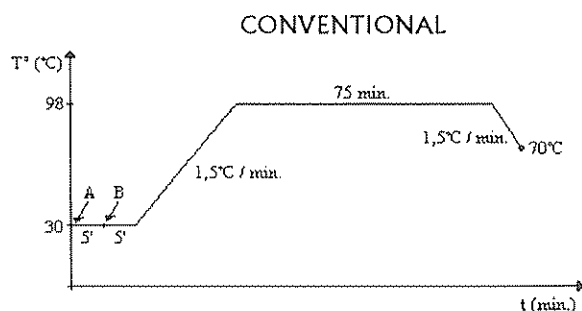
## 2.2 Wool/Acrylic/Alpaca yarn, dyed with metal complex dyes 1:2 for wool (1<sup>st</sup> bath) and cationic dyes for acrylic (2<sup>nd</sup> bath) (hank dyeing)

Hanks of worsted yarn, Nm 2/19, 50%Wool/30%Acrylic/20%Alpaca (intimate blend)

### DYEING CONDITIONS: 1<sup>st</sup> Bath

	RECIPES	
	CONVENTIONAL	LIPOSOME
<b>A – Auxiliaries:</b>		
Scourol S-520	1,0 g/l	-----
ECO TRANS W - 8814	-----	0,8 % o.w.f.
Protintex IWT	1,0 % o.w.f.	-----
Eulisin S	1,5 % o.w.f. (pH: 4,5 - 5,0)	1,5 % o.w.f. (pH: 4,5 - 5,0)
<b>B – ACIDOL Dyestuffs:</b>		
MTR Dark blue	3,5 % o.w.f.	3,5 % o.w.f.
R.B.: 35 l/Kg		

### DYEING DIAGRAM: 1<sup>st</sup> Bath

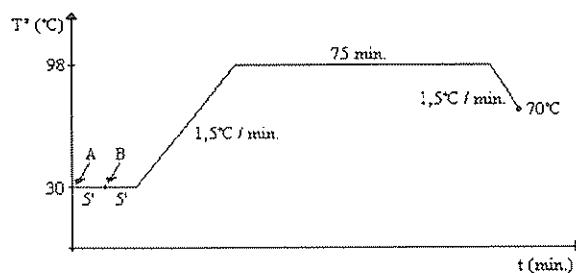


### DYEING CONDITIONS: 2<sup>nd</sup> Bath

	RECIPES	
	CONVENTIONAL	LIPOSOME
<b>A – Auxiliaries:</b>		
Protintex IWT	1,0 % o.w.f.	1,0 % o.w.f.
Vatel Sale	5,0 % o.w.f.	5,0 % o.w.f.
Eulisin S	0,5 % o.w.f. (pH: 4,5 - 5,0)	0,5 % o.w.f. (pH: 4,5 - 5,0)
<b>B – Dyestuffs:</b>		
ASTRAZON Blue 2RN	0,8 % o.w.f.	0,8 % o.w.f.
R.B: 35 l/Kg		

DYEING DIAGRAM: 2<sup>nd</sup> Bath

## CONVENTIONAL AND LIPOSOME



## RESULTS:

		CONVENTIONAL METHOD	LIPOSOME METHOD
COD 1 <sup>st</sup> initial bath (mg/l O <sub>2</sub> )		2681	1187
BOD <sub>5</sub> 1 <sup>st</sup> initial bath (mg/l O <sub>2</sub> )		573	371
COD 1 <sup>st</sup> final bath (mg/l O <sub>2</sub> )		2394	854
BOD <sub>5</sub> 1 <sup>st</sup> final bath (mg/l O <sub>2</sub> )		527	338
COD 2 <sup>nd</sup> initial bath (mg/l O <sub>2</sub> )		881	460
BOD <sub>5</sub> 2 <sup>nd</sup> initial bath (mg/l O <sub>2</sub> )		145	73
COD 2 <sup>nd</sup> final bath (mg/l O <sub>2</sub> )		893	432
BOD <sub>5</sub> 2 <sup>nd</sup> final bath (mg/l O <sub>2</sub> )		218	99
Tensile strength of undyed yarn (cN)		853±14	
Coefficient of variation (%)		5,9	
Tensile strength after dyeing (cN)		748±14	757±14
Coefficient of variation (%)		6,7	6,4
Colour fastness to washing (ISO 105 C06 A2S)	Colour change	4-5	4-5
	Staining (WO/PA/CO)	4-5 / 4-5 / 4-5	4-5 / 4 / 4-5
Colour fastness to water (ISO 105 E01)	Colour change	4-5	4-5
	Staining (WO/PA/CO)	4-5 / 4-5 / 4-5	4-5 / 4-5 / 4-5
1 <sup>st</sup> dyebath exhaustion (%)		97,9	98,1
Optical density of 1 <sup>st</sup> final bath (%)		0,3079	0,1931
2 <sup>nd</sup> dyebath exhaustion (%)		98,9	98,9
Optical density of 2 <sup>nd</sup> final bath (%)		0,0808	0,0723
Colour levelness		ACCEPTABLE	ACCEPTABLE
Costs (PTE/Kg): - Dyes and auxiliaries		133,20	115,49
- Electrical energy		3,30	3,17
- Thermal energy		93,45	80,5
Total		229,95	199,16

In what concerns colour levelness no difference was found when dyeing Acrylic/Wool by conventional and Liposome method. For Wool/Acrylic/Alpaca blend the appearance of small stripes has been noticed corresponding to small portions of yarn with a slightly darker shade in both processes, although with a more marked contrast in the Liposome dyed yarn. It is possible that this fault may be related to an uneven fibre blend.

The dyebath exhaustion is quite high in both processes, no significant differences in the data obtained for each one were found.

The polluting indices of 1<sup>st</sup> exhaustion bath as a result of wool dyeing with liposomes is about 53 to 64% lower than that found for the conventional dyeing in terms of COD and about 36 to 45% lower in terms of BOD<sub>5</sub>. Concerning the 2<sup>nd</sup> exhaustion bath from the acrylic dyeing COD and BOD<sub>5</sub> values are also lower in the Liposome dyeing procedure, the differences being about 52% for COD and 32 to 52% for BOD<sub>5</sub>.

The colour fastness levels are identical in both processes for Acrylic/Wool blend. For Wool/Acrylic/Alpaca blend a difference of a half step is observed in what concerns the staining on polyamide, the lower value corresponding to the Liposome dyeing. This difference was removed by a scouring after treatment of the dyed yarn.

The dyed yarn strength data using liposomes and the conventional method present no significant differences, although, a tendency to higher values is observed for Liposome dyeing.

The Liposome process allows a reduction of dyeing costs from 13% to 19%.

## **CONCLUSIONS**

Dyeing with liposomes is a feasible process at industrial scale for technical and economical reasons, provided that liposomes are available in the market at competitive prices, its use do not needing special attentions and offering good results for a large number of dyestuffs for pure wool and wool blends.

Dyeing with liposomes at 90°C reduce fibre damage during dyeing, obtaining higher values of tensile strength. A higher yield in the spinning of raw material dyed with liposomes was obtained.

The fastness levels of Liposome dyeings are similar to those of conventional dyeings. A slightly higher staining, concerning the fastness to water and a slightly higher change of colour concerning the fastness to washing were observed for some dyeings with liposomes (wool/polyamide blend, using 1:1 metal complex dyes and wool/acrylic/alpaca blend using 1:2 metal complex and cationic dyes). An improvement of colour fastness may be possible, in these cases, by raising the temperature from 90°C to 100°C at the final steps of the dyeing process or by a scouring after-treatment of dyed material.

The Liposome dyeing process is in all cases studied less pollutant than the conventional one. The Liposome dyeing process allows reducing significantly the treatment costs in comparison with the conventional process, even in special cases where a combination Liposome-retarding agent is needed to reduce the dyeing speed.

At the moment, with the Liposome in the market it is possible to guarantee satisfactory results in raw, top material and yarn dyeing. The dyeing temperature could be 10°C lower than in the conventional dyeing, the dye exhaustion obtained is in the same level of the conventional dyeing, and the environmental impact is lower. However, this Liposome does not favour dye migration to achieve good leveling of woven fabric without presence of synthetic auxiliaries. Design of new Liposome formulations is envisaged.